Characterization of the SiC and TiN whisker microstructure

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Five criteria for evaluating the whisker microstructure have been suggested, which include (1) proportion of “perfect” single crystals, (2) morphology and growth direction, (3) length, diameter and ratio of L/D, (4) the bulk and surface chemistries, (5) defects and their distribution. Three sets of commercially available SiC whiskers made in two countries and the TiN whiskers were characterized in terms of the above criteria.

1. Introduction
SiC and TiN whiskers are of considerable interest as reinforcements for metal or ceramic matrix composites. SiC whisker-toughened aluminium has received much attention because of its attractive mechanical and thermal properties. The addition of SiC whiskers to toughen and strengthen mullite, zirconia and silicon nitride is being pursued for high-temperature applications. However, the use of TiN whiskers is rather limited. Owing to a good match of thermal properties between TiN whiskers and zirconia matrix, ZrO₂ composite material reinforced with TiN whiskers is a good candidate for advanced heat engines, cutting tools, and for other applications.

The basic mechanism of strengthening in the composite is the load transfer to the whiskers as pointed by Rice [1]. The mechanical properties of matrix and reinforcing whiskers will certainly affect the strength of composites. Recently, work has been performed in order to reveal that not only the mechanical properties of the whisker bulk, but also whisker microstructure may change the strength and the fracture toughness of composites. The strength and toughness of aluminium–20 vol % SiC whisker composites were improved with whisker length through increasing the pull-out length of the whisker [2]. The possibility of improved pull-out resistance using a twinned SiC whisker for reinforcement should not be overlooked [3]. Defects present in SiC whiskers may degrade the mechanical properties of the whiskers as well as the composites [4, 5]. The chemical and morphological properties of SiC whiskers would be expected to affect the processing and mechanical properties of the resulting composites [6].

Silicon carbide whiskers are currently available from several manufacturers. The characterization of whiskers reported by manufacturers normally involved phase composition, the diameter and length, Young’s modulus, tensile strength, bulk compositions, etc., yet no thorough characterization of whisker microstructure has appeared in the literature. The intent of this work was to suggest five common factors for evaluating whisker microstructure, to present the main results for investigating SiC and TiN whiskers and thereby to understand better both the mechanisms of growth and the mechanical behaviour of the whiskers.

2. Experimental procedure
Three sets of SiC whiskers were used in this investigation. Two (labelled as C1 and C2) were made by the Institute of Metal Research, Academia Sinica and both were manufactured by a solid reaction method but treated by different procedures [7]. A third was a commercial product from Tokai Co., Japan (JTK). The TiN used was made by the Institute of Nuclear Energy Technology, Tsing Hua University, by chemical vapour deposition in the TiCl₄-N₂-H₂ system [8].

The phase composition of whiskers was examined by X-ray phase analysis. The morphology and microstructure of the whiskers were observed by analytical electron microscopy, JEOL 2000FX, and a high-resolution electron microscope, JEM 200CX, equipped with an ultra-high resolution pole-piece. Whiskers were easily dispersed; however, they seem to be rather thick for high-resolution imaging. Two kinds of composite reinforced by SiC whiskers with polymer and aluminium matrices, were obtained, and a cross-section of samples of whiskers was used in order to elucidate the transverse section shape and defect structures of the whiskers.

Bulk chemical composition analysis was carried out by energy dispersed spectrometry of X-ray micro-analysis (EDS) in transmission electron microscopy
(TEM) mode. Surface chemical composition analysis was performed in Riber Las 3000 and PEI 595 Auger electron spectrometers. The specimen used for surface analysis was prepared by impressing the whiskers into a soft indium plate. Thus the results obtained in surface analysis should be in the sense of statistics rather than an accurate measurement on a single whisker.

3. Results and discussion

The five criteria which are believed to be important for evaluating the whisker microstructure are:

1. the phase composition and percentage of "perfect" single crystals;
2. the morphology, direction and plane of whisker growth;
3. the diameter, length and ratio between length and diameter;
4. the surface and bulk composition;
5. defect type and distribution.

The results for the SiC and TiN whiskers will be reported in turn.

3.1. Crystal structure and percentage of "perfect" single crystals

Phase identification was based on comparison with standard X-ray diffraction data cards. It was ascertained that only the β-SiC or TiN phases were present in the C1, C2, JTK and TiN whiskers obtained, respectively. TiN is an interstitial compound of NaCl-type structure with lattice constant $a_0 = 0.424 \text{ nm}$. Fig. 1 shows a single TiN whisker with a straight rod shape and its selected-area electron diffraction patterns taken from different regions of the whisker. It can be seen that the electron diffraction patterns remain the same when moving along the whisker long-axis direction. The "perfect" whisker was defined as: (1) being a single crystal on the whole; (2) having a rather straight shape. About 50% TiN whiskers belong to the "perfect" group.

![Figure 1](A straight TiN whisker with single-crystal structure on the whole, and with a straight shape.)

![Figure 2](Variety of morphologies in the Cl and JTK SiC whiskers. Classified morphologies and the corresponding proportion of whiskers are also shown.)